SOLIDIFICATION, MICROSTRUCTURE AND MECHANICAL PROPERTIES OF MG-ND-GD-ZN-ZR CAST MAGNESIUM ALLOY WITH YTTRIUM, ERBIUM AND SAMARIUM ADDITIONS

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To my parents, wife, kids and my supervisor

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ABSTRACT

The thesis project an investigation the effect of alloying elements Yttrium, Erbium and Samarium addition on the solidification characteristics, microstructure and mechanical properties of as-cast Mg-Nd-Gd-Zn-Zr magnesium alloy. The future automotive and aerospace industries will experience tremendous progress if magnesium alloys become an integral part of their production unit mainly because of their light weight. The main limitation to progress in this direction is paucity in the mechanical properties of magnesium alloys. Rare earth elements have been used as alloying elements to improve mechanical properties and introduce a new Mg-RE-Zn-Zr alloy with modified structure and strength. In addition, this study demonstrates the addition of RE to specific quantities that could be considered as major alloying elements that could lead to RE application extension limits. 0.1, 0.25, 0.5, 1, 1.5, 2 and 2.5 wt.% of Y, Er and Sm were added separately to Mg-Nd-Gd-Zn-Zr magnesium alloy. Thermal analysis was examined using CA-CCA. OM, SEM/EDS and XRD were used to investigate the microstructure of alloys, and the mechanical properties investigated include tensile and hardness tests. The results revealed that as Y level reached 0.25 wt.%, the solidification time was reduced to 27.28 s, the grain size decreased by 42.5%. Addition of 0.5 wt. % of Er caused a decrease in the solidification time of about 7.5 % which led to a decrease grain size of about 46 %. Furthermore, solidification time was reduced to 33.56 s which led to grain size reduction by 41.7 % as addition of 1.5 wt.% Sm. UTS and YS were improved by 37.3 % and 19.6 % respectively through addition of 0.25 wt. % of Y. Moreover, UTS increased by 21.4 % and 7.5 % for YS at 0.5 wt.% of Er. Addition of 1.5 wt. % of Sm led to increase UTS and YS by 14.5% and 15.9% respectively. In addition, the hardness value of base alloy was also recovered. The additives had a significant influence on the setting time, led to the improvement of the microstructure and mechanical properties, so that the magnesium alloy Mg-Nd-Gd-Zn-Zr was developed.

ABSTRAK

Projek tesis ini menyiasat kesan penggabungan elemen Y, Er dan Sm pada ciri pemejalan, struktur mikro dan sifat mekanik aloi magnesium Mg-Nd-Gd-Zn-Zr. Industri automotif dan aeroangkasa akan datang akan mengalami kemajuan besar jika aloi magnesium menjadi sebahagian daripada unit pengeluaran mereka terutamanya kerana berat ringan mereka. Batasan utama untuk maju ke arah ini adalah kekurangan dalam sifat mekanik aloi magnesium. Unsur-unsur nadir bumi telah digunakan sebagai unsur-unsur aloi untuk memperbaiki sifat-sifat mekanik dan memperkenalkan aloi Mg-RE-Zn-Zr yang baru dengan struktur dan kekuatan diubahsuai. Di samping itu, kajian ini menunjukkan penambahan RE ke kuantiti tertentu yang boleh dianggap sebagai unsur pengaloi utama yang boleh membawa kepada had pelanjutan permohonan RE. 0.1, 0.25, 0.5, 1, 1.5, 2 dan 2.5 wt% Y, Er dan Sm ditambah secara berasingan kepada aloi magnesium Mg-Nd-Gd-Zn-Zr. Analisis haba telah diperiksa menggunakan CA-CCA. OM, SEM / EDS dan XRD digunakan untuk menyiasat struktur mikro aloi, dan sifat-sifat mekanik yang disiasat termasuk ujian tegangan dan kekerasan. Keputusan menunjukkan bahawa tahap Y mencapai 0.25%, masa pemejalan dikurangkan kepada 27.28 s, saiz bijian menurun sebanyak 42.5%. Penambahan 0.5 wt. % dari Er menyebabkan penurunan dalam masa pemeimbangan kira-kira 7.5% yang menyebabkan penurunan saiz biji kira-kira 46%. Tambahan pula, masa pemejalan dikurangkan kepada 33.56s yang membawa kepada pengurangan saiz bijian sebanyak 41.7% sebagai tambahan 1.5 wt.% Sm. UTS dan YS masing-masing meningkat sebanyak 37.3% dan 19.6% melalui penambahan 0.25 wt. % daripada Y. Selain itu, UTS meningkat sebanyak 21.4% dan 7.5% untuk YS pada 0.5%% daripada Er. Penambahan 1.5 wt. % dari Sm mendorong peningkatan UTS dan YS masing-masing sebanyak 14.5% dan 15.9%. Di samping itu, nilai kekerasan aloi asas juga pulih. Aditif mempunyai pengaruh penting pada masa penetapan, menyebabkan peningkatan struktur mikro dan sifat mekanik, supaya aloi magnesium Mg-Nd-Gd-Zn-Zr dikembangkan.

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LIST OF SYMBOLS AND ABBREVIATION

Υ_{SL}	solid-liquid interfacial energy
ΔG_V	Gibbs free energy
r	radius
θ	wetting angle
CA-CCA	Computer Aided -Cooling Curves Analysis
ASTM	American Society Testing Materials
BCC	Body Center Cubic
FCC	Face Center Cubic
HCP	Hexagonal Close Packed
SEM	Scanning Electron Microscope
EDS	Energy-Dispersive X-ray Spectroscopy
XRD	X-Ray Diffraction
UTS	Ultimate Tensile Strength
YS	Yield Strength
El	Elongation
s	Second
T	Temperature
T_N	Nucleation temperature
T_G	Growth temperature
t_N	Nucleation time
t_G	Growth time
ΔT	Solidification temperature
Δt	Solidification time
Ts	End of solidification
T_{DCP}	Coherency temperature
t_{DCP}	Coherency time

CHAPTER 1

INTRODUCTION

1.1 Research background

The current challenge involves the increasing use of light alloys in high-technology materials, with the aim of both reducing mass and energy saving. The related typical application areas include vehicle construction, aeronautics and the space sector, along with the mechanical engineering[1]. Moreover, the increasing concerns on environmental protection and sustainable economic development draw a great deal of attention in reducing greenhouse gas emissions[2]. Protecting the atmosphere and reduction of CO2 traffic emissions grew in importance in the social discussion. Reduction in fuel consumption for motor vehicles therefore had a significant influence on CO2 emissions. Reduction in vehicle mass leads to reduction in fuel consumption[3]. Therefore, weight reduction has become an important step to make effective use of fuel cells and hence lower energy consumption.

Reduced weight of products can be attained by many approaches such as redesign, removal of unnecessary parts, reducing thickness and selection of light materials. For such reasons, much effort has been spent on developing lighter materials, such as composites, and aluminum and magnesium alloys for automotive, and aerospace applications[4]. Magnesium is the 6th most plentiful component in the world's covering and is the lightest of every single auxiliary metal with a high specific stiffness. This is one of the prime reasons car producers are in a journey to supplant denser materials with magnesium (Mg) based materials. In any case, poor formability (ductility) and secondary processing induced crystallographic asymmetry due to the hexagonal closed pack (HCP) crystal structure represent the major limitations of Mg[5]. It has limited slip systems and the activation of non-basal slip is difficult at room temperature, thereby limiting the ductility. This limitation is being overcome with the development of new magnesium based alloys[6].

The fact that they are still the lightest metal alloys commercially available to engineers makes them the most attractive materials for light weight applications[7]. Magnesium alloys are attractive as light-weight structural materials for space industries and automobile due to their low density, high specific strength, high damping capacities and good casting properties[8]. Magnesium alloys can be divided into cast and wrought magnesium alloys. Main commercial magnesium alloys include the AZ series (Mg-Al-Zn), AM series (Mg-Al-Mn), AE series (Mg-Al-RE), EZ series (Mg-RE- Zn), ZK series (Mg-Zn-Zr), and WE series (Mg-RE-Zr). Statistically, more than 90% of the magnesium alloy structural components are produced by casting process[9].

Mg–RE (rare-earth elements, such as Gd, Y, Ce, Nd, mischmetal, etc.) alloys have been given tremendous attention due to their high specific strength at room and elevated temperatures as well as their excellent creep resistance[10]. Magnesium-rare earth-zinc-zirconium alloys show good casting property because the presence of the rare earth elements promotes formation of relatively low melting point eutectics that improve fluidity and tend to prevent microporosity. The properties of Mg-RE alloys are enhanced by adding zirconium to refine grain size and further increase in strength occurs if zinc is present as well[4].

Strengthening is the ability of alloy to plastically deform depends on the ability of dislocations to move. Several strengthening mechanisms contribute to the improvement of mechanical properties, such as solution strengthening, cold working, precipitation of phase during solid solution decomposition, grain refinement and precipitation of other phases[11]. Grain size strengthening efficiency in Mg alloys is much higher than that in Al and other alloys. Fine grain size can result in structural uniformity and enhance the mechanical properties, hence improving the service performance of the products[12]. During the solidification, the grain refinement can be achieved by two major mechanisms: promoting the heterogeneous nucleation and restricting the grain growth[13]. A reduction in grain size serves to enhance mechanical property as formulated by the well-known Hall-Petch strengthening, which is a method of strengthening materials by changing their average grain size[14].

Solid solution strengthening comes from atoms in the solution, which change the lattice parameters and binding forces of Mg alloys. The solution atoms interact with dislocations and twinning in the Mg alloys, which strengthen the alloys. Second phases strengthening is the presence of second phases which will strengthen a material by blocking dislocation motion[15]. It is based on the observation that grain boundaries impede dislocation movement and that the number of dislocations within a grain have an effect on how easily dislocations can traverse grain boundaries and travel from grain to grain. The solid solubility of rare earth element is high in magnesium. Rare earth atoms dissolve in the magnesium matrix, enhance the binding force of atomic ask, and distort the matrix lattice[16].

The RE could be divided into light RE (La, Ce, Pr, Nd, etc.) and heavy RE (Gd, Tb, Dy, Ho, etc.). Magnesium alloys with heavy RE such as Mg–Gd-based alloys with Gd addition over 10 wt% have been extensively studied for their potential in achieving higher strength and better creep resistance. Among Mg–light RE binary alloys, Mg–Nd alloys show a higher strength for a high equilibrium solid solubility. Meanwhile, it was reported that Nd addition in Mg–Gd-based alloys could improve the mechanical properties[17]. The periodic table is organized so scientists can quickly discern the properties of individual elements such as their mass, electron number, electron configuration and their unique chemical properties. Rare earth elements are a group of seventeen chemical elements that occur together in the periodic table. The group consists of Yttrium and the 15 lanthanide elements (lanthanum, cerium, praseodymium, neodymium, promethium, samarium, europium, gadolinium, terbium, dysprosium, holmium, erbium, thulium, ytterbium, and lutetium). The rare earth elements are all metals, and the group is often referred to as the "rare earth metals." These metals have many similar properties and that often cause them to be found together in geologic deposits. They are also referred to as "rare earth oxides" because many of them are typically sold as oxide compounds[18].

The RE element has solid solution strengthening and precipitation strengthening effect, it can significantly improve the tensile properties and casting performance of magnesium alloy without jeopardizing the advantages of magnesium such as low density, high specific strength and excellent rigidity[19]. Rare earth-enabled products and technologies help fuel global economic growth, maintain high standards of living, and even save lives [20]. Alloying magnesium with rare-earth elements (RE), is used to develop light construction alloys. Generally, RE elements in Mg have relatively high solubility decreasing significantly with decreasing temperature[21]. At present, the rare-earth elements added into magnesium alloy are generally divided into two categories: one is the elements with small solid solubility such as Ce, Pr element and so on; another is the elements with large solid solubility such as Y, Nd, La, Sm element etc.[22].

Recent studies highlighted that some rare earth (RE) metals, such as Yttrium (Y), Neodymium (Nd), Erbium (Er) are very effective elements to improve the mechanical properties of Mg alloys. Varying amounts of alloying elements added to the magnesium result in changes to mechanical properties such as increased tensile strength, creep resistance, thermal stability or corrosion resistance[21]. Yttrium is a chemical element with symbol Y and atomic number 39. Y is a soft, silver-metallic, lustrous and highly crystalline transition metal and it is a silvery-metallic transition metal chemically similar to the lanthanides and has often been classified as a "rare-earth element". Erbium is a chemical element with symbol Er and atomic number 68. Erbium is a soft, malleable, lustrous, silvery metal. It is very stable in air; it reacts very slowly with oxygen and water and dissolves in acids. Samarium is a chemical element with symbol Sm and atomic number 62. It is relatively stable at room temperature in dry air, but it ignites when heated above 150 C and forms an oxide coating in moist air.

Solidification in commercial casting processes occurs under equilibrium conditions. Solidification also is a crucial stage of casting production. Fundamental knowledge of solidification characteristics and of the resulting microstructure is a prerequisite to understand the mechanical behavior of the component. Controlling the as-solidified microstructure often provides the alloy designer with the greatest influence over the final alloy performance and should be considered paramount to the intelligent, rapid design of improved alloys. Much of this knowledge is yet to be discovered, therefore, the researchers are working on it with appropriate research equipment designed for studying solidification, microstructure and microstructure evolution[23]. The solidification microstructure on any cast alloy will depend on the alloy content and the casting process. These casting processes are characterized by intermediate to cooling rates and decline temperature gradients. Practically all magnesium for structural applications is high pressure die cast. Most commercial magnesium alloys are primary eutectic alloys, implying that solidification starts by nucleation and growth of primary α-Mg, followed by dendritic growth of secondary phase and ends by eutectic reactions for ternary phase[24, 25]. The slow cooling rates encountered in sand casting tend to produce coarse microstructures that have low ductility. To improve ductility the grain size needs to be decreased by the addition of grain refiner.

Solidification is the growth to a stable phase of material from the unstable liquid phase. The key solidification processes that control the final microstructure are the initial nucleation events, the growth of these nuclei into primary dendrites and finally eutectic solidification[26]. The knowledge of solidification and resultant microstructure is fundamental to the alloy development process. The solidification of alloys has a number of variables, such as their components and relative amounts, which lead to formation of phases across certain temperature ranges; once these phases are created, the overall microstructure of the alloy is formed. A phase diagram displays information about the control of the phase structure of a system. Most commercial magnesium alloys are primary eutectic alloys, implying that solidification starts by nucleation of magnesium, followed by dendritic growth and ends by eutectic reactions. In general, three main lines outline the boundaries of a phase diagram: liquidus, solidus and eutectic. The liquidus separates the liquid and solid phases. Upon passing through the liquidus the primary phase forms. Formation of the primary phase will continue to occur until eutectic or solidus line, below which only the solid phase exists. The eutectic line is isothermal and characterizes the equilibrium between phases formed[27]. A series of phase transformations occur during the solidification of a casting from the liquid state. In simple binary alloys such as Al–Si or Mg–Al the primary α-phase nucleates first and, as the temperature falls, the α-phase will grow until the eutectic temperature is reached at which point the eutectic nucleates consuming the remaining liquid. In ternary alloys such as the Al–Si–Cu alloys, a ternary eutectic will also form before solidification is complete[28]. All the commercial magnesium alloys start forming magnesium solid solution at the early stage of solidification which is denoted as (α-Mg). The nucleation of the primary phase is sometimes controlled by the addition of grain refiners[23].

The classic nucleation theory is a phenomenological theory that assumes that clusters of atoms or molecules form spontaneously in the matter undergoing transformation[29]. The classical nucleation theory (CNT) is the first approach on the nucleation theory. The formulation of CNT relies on equilibrium thermodynamics and the use of macroscopically determined properties. To form a small solid sphere with radius r in a super cooled liquid, the change in Gibbs free energy can be expressed as

$$\Delta G_r = [-(4/3)*\pi r^3 \Delta G_V + 4\pi r^2 \Upsilon_{SL}]S(\theta) \qquad (1)$$

Where Υ_{SL} is the solid-liquid interfacial energy, ΔG_V is the Gibbs free energy difference per unit volume between solid and liquid phases at the same temperature, (θ) is the factor in terms of the wetting angle θ:

$$(\theta) = (2 + \cos\theta)\,(1 - \cos\theta)^2/4 \qquad (2)$$

The two main types of nucleation are homogeneous nucleation, where the new phase is in a uniform substance, and heterogeneous nucleation, where nucleation occur a pre-existing substrate. In fact, homogeneous nucleation is the most difficult kinetic path for crystal formation. Homogeneous nucleation considers a given volume of liquid at temperature below the liquidus of the metal. In classical nucleation theory, heterogeneous nucleation comprises the thermodynamics and the kinetics of the formation of the nuclei of a new phase on the surface of a foreign substrate[13]. The growth of magnesium dendrites occurs according to the usual principles of primary phase solidification (α-Mg). Solute elements with a distribution coefficient less than unity are rejected ahead of the dendrites and this can cause changes in the microstructure[26]. Dendrites of magnesium grow at angles between dendrite arms of 60^0 . This process is characterized by very high gate velocities, and also high cooling rates, which tend to fragmentize and refine the dendrites[24].

During solidification of a eutectic melt several solid phases appear at the same time while no melt will be left. In the case of binary eutectics two phases are created. They can be arranged as lamellae or as fibers in a matrix. Which pattern will be created depends on the eutectic composition[30]. Magnesium forms eutectic systems with a wide range of alloying elements and the microstructures of the common casting alloys, ZRE1 and AM50 as well as recently developed alloys such as ZA85, contain small amounts of eutectic. Pure magnesium is very weak and low to moderate levels of alloying provide solid-solution strengthening and strengthening from the presence of massive second-phase particles formed during eutectic solidification[26]. Modification of the eutectic phase greatly enhances the mechanical properties of the alloy[24]. The occurrence of each type of eutectic depends on the zinc contents and the cooling conditions[31]. During eutectic transformation, atomic diffusion causes distribution of system components to form the eutectic microstructure. Cooling either side of the eutectic isotherm results in transformation of the morphology and a mixture of micro constituents of primary and eutectic phases[27]. In the case of ternary eutectics phases are created. However, in reality ternary eutectics are much more complex[32].

However, instances where the ternary systems contain intermetallic phases which are different from any which occur in the relevant binary systems. Although the occurrence of ternary compounds cannot as yet be confidently predicted, in magnesium alloys the formation of these compounds is affected strongly by two factors, which are related to the electropositive nature of magnesium and to the relatively large radius of the magnesium atom, respectively[33]. Consequently, the activation energy (ΔG^*) that must be overcome to produce stable nucleation is lower, and so ternary phase is easy to nucleate. In this equation, ΔG refers to free energy difference and the driving force of the transformation, δ_{SL}, is the interfacial energy of solid liquid two phase and the resistance of the phase transformation[34].

$$\Delta G^* = 16\delta^3_{SL} / 3\Delta G^2 \qquad (3)$$

The two main stages in solidification are nucleation and grain growth. Thus, in order to achieve refinement of microstructures, we can either enhance nucleation or restrict grain growth. In the previous research studies, a number of grain refinement technologies have been developed and applied to magnesium alloy castings. The phenomenon of solidification takes an important role in various fields since it causes great impact in time taken for production, quality of product cast and the quantity of material used for the production.

The thermal analysis technique is a well-established technique, both in ferrous and nonferrous industries to assess the quality of the melt prior to casting. However, obtaining the appropriate microstructure of a standard cup does not guarantee that the microstructure is correct in real parts which may solidify in a very various cooling rates. The solidification characteristics can be investigated through various thermal analysis techniques. There are standardized techniques such as differential thermal analysis (DTA), thermo gravimetric analysis (TGA), differential scanning calorimetry (DSC) and computer aided cooling curve analysis (CA-CCA). Due to its ease of use and low-cost, computer aided cooling curve analysis (CA-CCA) is much more appropriate for industrial applications compared to other techniques and computer aided cooling curve analysis (CA-CCA) method has been

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